



Heavy metals pollution potentials in the National Iron Ore Mining Company, Itakpe

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ABSTRACT

Heavy metals pollution potential in National Iron Ore Mining Company, Itakpe was investigated. Two mining sites located at the east mining pits such as M_3O , which is 370 m above sea level and M_2O , which is 350 m above sea level were studied. Sequential extraction techniques was utilised to examine the distribution effect of the heavy metals pollution potential on the environment. Twelve representative (six-soil, two-sediment, two-plant and two-water) samples were collected, pre-treated and prepared for this study. The atomic absorption spectrometer was used to analyse the concentration of the metals after the sequential and single-stage extractions were determined. Results showed that Chromium, Arsenic, Cadmium and Copper are more bioavailable in the study area than Lead and Iron. This findings indicate that human, animals and plants are exposed to toxic elements (metals and metalloids).

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1. Introduction



Several iron ore deposits have been discovered in Nigeria since 1904. These deposits include magnetite, hematite, siderite-goethite and goethite grades [1]. The projected reserve is over 3 billion metric tonnes; their use in steel and iron plants will cut down the cost of importation. This could enhance foreign exchange, augment revenue generation as well as indigenous technology transfer for agriculture and military defence, and create employment.



The Itakpe iron ore deposit is located northeast of Okene, Kogi state. This ore deposit is presently the most highly explored ferrous deposit in Nigeria. Its estimated

reserve is more than 300 million tonnes but with established reserve of 200 million tonnes [2]. In the quest for industrialisation, the government established a 1.3 million metric tonnes per annum steel plant based on the blast furnace process at Ajaokuta, in Kogi state. The plant intended to use iron ore from the National Iron Ore Mining Company (NIOMCO), Itakpe, 60 km by rail from Ajaokuta Steel Company; as one of its primary raw materials. Additionally, NIOMCO will supply iron ore concentrate (super concentrate) to the Delta steel company, Aladja, Delta state. Iron ores are available in commercial quantities in Anambra and Edo states. However, the most important iron ore

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areas are in Itakpe, Kogi state and Jebba, Kwara state [4].

The mining and beneficiation processes at NIOMCO consist of solid waste disposition containing organic matter. In most cases, such depositions contain contaminants such as heavy metals that have effects on the ecosystem [5]. These metals, if available in high concentration, are toxic to aquatic life, plants as well as human health. The influence of these toxic chemicals on the ecosystem is of intense interest due to the extensive usage of a broad range of metals for both industrial and domestic applications. The clinical attributes of different metals that contain industrial effluents, which comprise a main source of metallic pollution of the hydrosphere as described in [6]. The effect of these metal ions on mammalian systems is as a result of chemical reactivity of the ions with membrane system, enzymes and cellular structural proteins. In particular, the metals (including Arsenic, Cadmium, Chromium, and Lead) have caused acute and chronic poisoning in humans and animals [6].

In [7] chemical mutations of coal fly ash treated with different compounds was undertaken. The objective was to compare the adsorption of Pb(II) and Cd(II) ions from water solution on treated fly ash. Experimental results showed that the improved coal fly ash enhanced the uptake of Pb(II) and Cd(II) ion from contaminated waters. The chemical speciation heavy metals in mining wastewater was reported in [8]. The results showed although patterns of the elements (Zn, Cu, Pb, Cr, As and Sn) were discovered in the reducible fraction Pb and Sn were mainly linked with the organic fraction.

The aim of this paper is to investigate the pollution potentials of heavy metals in the National Iron Ore Mining Company, Itakpe.

2. Materials and Methods

2.1. Site Description

The National Iron Ore Mining Company, Itakpe (longitude 6°36'1 E and latitude 7°36' N) is located in the north-central part of Nigeria; 60km by rail from Ajaokuta Steel Company Limited [4]. The company, which is concerned with the mining and beneficiation of iron ore concentrates is made of three major sections: two mining sites (the east mining pits M₃O, which is 370 m above sea level and M₂O, which is 350 m above sea level) and the processing/smeltering section where the beneficiation plant is situated. Behind the beneficiation plant is river Pom-Pom where

the effluent coming from beneficiation of the iron ore concentrates is dumped. Physical observation of the entire NIOMCO vicinity shows big and small rocks, stones and concretes, shiny dark brown soils and some clear sandy soils. Animal dung and vegetation are also seen within close range to the mining sites [7].

2.2. Sampling

As at the time of sampling, the company has stopped operation for about a year. Sampling was done on 20th January 2018, at different sites within the Itakpe iron ore mining complex. Twelve representative samples comprising six soil samples, two sediment samples, two plant samples and two water samples were collected. The elevation as well as the longitudes and latitudes of the different sampling were obtained using an Etrex (IV) channel GPS. The distance between the sampling points at each area of the complex is 500 m. Table 1 shows the normal content range and maximum acceptable limits of heavy metals in soil while Fig. 1 shows the studied plant samples.

2.3. Pre-Treatment and Preparation of Samples

Soil and Sediments: These samples were collected within the depth of 5 – 15 cm into clean polythene sample bags. The collected samples were air-dried for seventy-two hours at the ambient temperature range of 28°C ≤ T ≤ 32°C. The particle size distribution of the samples was done using stainless sieves of diameters 0.002 mm, 0.00315 mm and 0.01 to obtain the percentage values of the different components of the samples. The different components obtained are sand (>0.00315 mm and < 0.01 mm), silt (>0.002 mm and < 0.00315 mm) and clay (<0.002 mm). The pH of the samples was determined [10] by the use of a glass electrode linked to a pH meter (Kent EIL, 7020). 10g of the samples were collected in 50ml distilled water stirred for 10 minutes. The resulting solution was allowed to settle for 1 hour but randomly

Table 1 Normal content intervals and maximum acceptable limits of heavy metals in soil [9]

Chemical element	Normal content interval (mg/kg)	Maximum allowable limits (mg/kg)
Cd	0.1 -1.0	3
Co	1 – 10	50
Cr	2 – 50	100
Cu	1 – 20	100
Ni	2 – 5	50
Pb	0.1 – 20	100
Zn	3-50	300

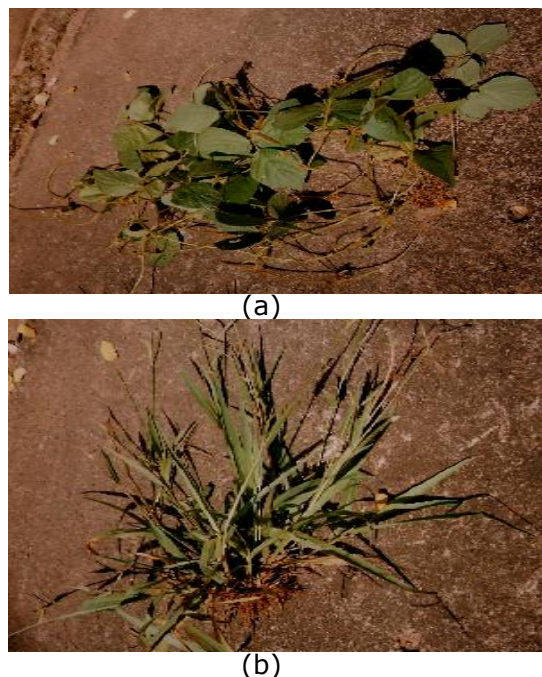


Fig. 1 Plant samples (a) *Calopogonium Mucunoides* (Calopo) (b) *Paspalum Scrobilatum* (Ditch millet)

stirred before obtaining the pH value.

The moisture content: The air-dried sample was homogenized (pulverization) using the electronic crusher (Herzog D4500, Osnabruck, Type: HSM100, Fabr. Nr. 62B/414) and sieved using the 0.002 mm diameter sieve. The (<0.002 mm) was used as the sample. 50g of the air dried sample was weighed into a previously cleaned dried and weighed stainless plate. This was charged into the oven at 105°C for 4 hours; with continuous weighing till constant weight (W₂). The difference between the initial weight and final/constant weight; divided by the 50g multiplied by 100 is the percentage of moisture content.

The organic matter and ash content in the soil and sediment samples were established by the ignition of a known weight of the sample using a clean, dry and weighed crucible in a muffle furnace at 550°C for one hour. The crucible together with the ash was cooled and weighed after the ignition. The percentage loss on ignition was considered as the total organic matter (TOM) [10]; while the percentage of ash is regarded as the ash content.

$$\text{TOM} = \frac{[(W_0 + W_1) - W_2]}{W_1} \times 100 \quad (1)$$

$$\text{Ash Content} = \frac{(W_2 - W_0)}{W_1} \times 100 \quad (2)$$

Where, W₀ is the weight of crucible before ignition, W₁ is the initial weight of the samples, and W₂ is the weight of crucible and sample after ignition.

After these samples were weighed, the soil and sediment samples were pulverized using the electronic crusher (Herzog D4500, Osnabruck; Type: HSM100, Fabr. Nr. 62B/414) and sieved with the 0.002 mm sieve, (the <0.002 mm fraction) were kept and used as the samples for both sequential and total extractions followed by the analysis of the extracted supernatant with the 210VGP Buck Scientific Atomic Absorption Spectrophotometer [11].

(a) *Water*: The water samples collected were directly transferred into clean plastic bottles by submerging and opening the bottles beneath the water surface. These samples were fixed with 2 ml of 1M HNO₃ (pH of 2-3) immediately at the point of collection. 100ml of the fixed sample was digested on the hot plate in the laboratory with 1 ml of 1M HNO₃ and 1ml of 1M HCl at 100°C till the volume was reduced to about 30 ml. The solution was cooled and filtered with Whatman number one filter paper and washed with deionized water into a 50ml volumetric flask. The filtrate was filled up to the mark with deionized water and was analysed with Atomic Absorption Spectrophotometer model 210VGP, Buck Scientific Incorporated USA [11].

(b) *Plants*: The plant samples were collected by gently uprooting the seedlings to avoid cutting off the roots; into clean polythene bags [7]. The plant seedlings were first identified at the Federal University of Technology, Akure. They were sun-dried at an ambient temperature range of 27°C ≤ T ≤ 31°C for a week and oven-dried at a temperature of 105°C for forty-eight (48) hours. The dried plant seedlings were homogenized using the Excella model of the Mixer Grinder (Marlex ® 230 V, made in India), sieved with 0.002 mm diameter sieve; (<0.002 mm fraction) was taken as the plant sample and extracted using the Single-stage extraction method (Conc. HNO₃, HClO₄ and HCl). The extracted supernatant was analysed with the 210VGP Buck Scientific Atomic Absorption Spectrophotometer.

3. Detailed Procedure of Sequential Extraction

Sequential extraction is refers to the identification and quantification of the diverse distinct species, phases or forms that the metals occur [12]. It is sometimes referred to as Speciation, Fractionation or Selective extraction. The experiment was carried out on each sample of soil and sediment in triplicates (n=3). The initial weight of each sample taken

was 5g. Table 1 contains a detailed representation of the research conditions and the compositions of extracting solutions of Tessier Sequential extraction scheme.

Step 1: The soil and sediment samples were transferred to a 250ml conical flask and 25ml of 1.0M MgCl_2 solution with a pH of 7 was added. The resulting mixture was stirred for 30 minutes at ambient temperature of 27°C at a speed of 200rpm (using a horizontal rotary mechanical shaker) [13]. Centrifugal method, at 3000rpm for 5 minutes, was used to separate the extract from the solid residue. Thereafter, the supernatant liquid was poured into a clean polyethylene container. The liquid was diluted to 50ml and used for the measurement of the metals. The residue was washed using deionized water (20 ml) and centrifuged for 15 minutes, and the supernatant was discharged without the solid residue that was used for the next step.

Step 2: The residue was treated with 25ml acetate buffer – $\text{CH}_3\text{COOH}/\text{CH}_3\text{COONa}$ at pH 5; shaking for one (1) hour and centrifuging. The extract was separated as in step one and used for measurement of metals. The residue was washed as in step one and kept for the next step.

Step 3: The residue obtained from the previous stage was treated with 25ml of 0.04M $\text{NH}_2\text{OH}.\text{HCl}$ in 25% (v/v) CH_3COOH ; digestion was done for two hours on a hot plate at 96°C with random mixing. The extract was removed as in step one and used for measurement of metals. The solid residue was washed and kept for the next step.

Step 4: To the residue obtained from step 3, 15 ml aliquot of 30% H_2O_2 (adjusted to pH value of 2 with HNO_3) were added and heated or digested at 85°C for one hour. The content of the flask was evaporated to about (1-2ml). A second aliquot of 10 ml of 30% H_2O_2 (adjusted to pH value of 2 with HNO_3) was added and the content of the flask was evaporated again at 85°C for one hour to near dryness. After vaporization of the solution, the cooled residual 10ml of 3.2M $\text{CH}_3\text{COONH}_4$ in 20% (v/v) HNO_3 was added and stirred continuously for 30min. Thereafter, the supernatant was diluted to 50ml, used for measurement of metals while the solid residue was washed with 20 ml of deionized water and reserved for the final step.

Step 5: The residue obtained from step 4 was treated with 20 ml mixture of HNO_3 and HClO_4 acids in the ratio 2:1. The flask was covered with a watch glass, and heated at 90°C for 1 hour. Then, the content was evaporated to dryness. 15 ml of HCl was

added to the dry cooled residue in equal ratio; and then heated for 5mins. Residual metals were measured in the filtrate. Blank extraction was performed for each set of analysis using the same reagent.

3.1. Total Metal Content

The total metal content of the observed samples was extracted using the Single-stage extraction method, which involves extraction with concentrated HNO_3 , HClO_4 and HCl . The conditions are the same as that of step 5 of the sequential extraction method. The experiment was done in triplicates ($n=3$).

Procedure: 5g of the samples were weighed in a 250ml conical flask and wetted with 5ml of concentrated. HNO_3 acid and 2.5 ml of concentrated HClO_4 acid. The flask was covered with a watch glass and heated on a heating plate (90°C) for an hour. After evaporating the flask content to dryness, 15ml of HCl solution (1:1) was added to the cooled dry residue and heated again as before. The flask content was hot-filtered by a medium quantitative filter paper, decanting sediment traces with the help of hot 1% (v/v) HCl . The obtained supernatant was analysed for Cr, As, Cd, Pb, Cu and Fe using the Buck Scientific Incorporated USA 210VGP model of Atomic Absorption Spectrophotometer (AAS). The reagent blank was simultaneously run.

3.2. Reagents

The reagents used are acetate buffer, magnesium chloride, hydroxylamine hydrochloride, 30% solution of H_2O_2 , acetic acid, nitric acid, ammonium acetate and perchloric acid. These reagents are either Aldrich or Fluka analytical grade. Diluted working solution was prepared using the corresponding final medium for each of the steps of the sequential extraction procedure. Calibrations and reagent solutions were made using double deionized water from Tisco Company Akure. All plastic and glass wares were soaked in 5% (v/v) HNO_3 for a minimum of 24 hours and rinsed with deionized water before use [13].

3.3. Instrumentation and Analysis

The concentrations of metals (Chromium, Arsenic, Cadmium, lead, Copper) and in the supernatants (after sequential and the single-stage extractions) were determined by an AAS (Buck Scientific Incorporated, USA 210VGP model) with a yellow fuel-rich air/acetylene flame of temperature 230°C . The burner height is 3 mm. All analytical determinations

were performed in triplicate. Description of the instrument calibration, and conditions for estimation the heavy metals in soil, sediment, plant and water samples are available in [14], [15]. Other instrumental parameters were given in Table 2. The results of the metal concentrations were corrected using that of the reagent blanks. Results were calculated per dry weight of samples.

3.4. Validation of the Analytical Method Used

The sequential extraction results were verified by comparing the total amount of metals extracted by the reagents during the extraction process with the results of the total digestion [8]. The percentage recovery of the sequential extraction was computed using the equation:

$$\text{Rec} = \frac{\sum_{n=1}^5 \text{Conc. } F_n}{\text{Conc. Total Digestion}} \times 100 \quad (3)$$

Where F_1 is the carbonate/acid-soluble fraction, F_2 is the extraction using 1M MgCl_2 , pH 7, 0.5 hours, 20° C, F_3 is the oxides, F_4 is the organics and F_5 is the residual increased while the F_1 is reduced.

3.5. Appraisal of Contamination / Pollution Levels

The heavy metal contamination level of the studied soil and sediments samples were

determined with the Dutch system of calculation pollution evaluation method [18]. In this system mathematical equations are used to establish the reference values specific to each sample, considering the organic matter and clay content of less than 0.002 mm. This is because these constituents directly influence the characteristics (physical and chemical) of the soil. This method permits calculations of the contamination/pollution (C/P index) to place a sample within the pollution range of the soil range, and is deduced as:

$$C/P \text{ index} = M_v / T_c \quad (4)$$

Where M_v is the measured value is the actual value of heavy metal concentration obtained from chemical analysis in mg/kg and T_c is the calculated target value in mg/kg as given in the DPR conversion formula as follows:

$$T_c = \frac{T_{st} (A + B + \% \text{Clay} + C \times \% \text{Organic matter})}{A + B \times 25 \times 10} \quad (5)$$

Where T_{st} (mg/kg) is the standard or reference target value; also denoted as T_v , and A , B and C are factors from the Detailed Project Report (DPR) formula, which vary according to each metal.

The DPR target and intervention values of metals for a standard soil/sediment is shown in Table 3.

Table 2 AAS instrumental parameters [16], [17]

Metals	Wavelength (nm)	Band pass (nm)	Lamp type	Relative sensitivity	Detection limit (µg/ml)	Optimum working range (µg/ml 200-800Abs)	Flame Type
Cr	357.9	0.5	HCL	1.0	0.002	2-15	Height Critical
	428.9	0.5	HCL	7.0		15-60	
As	193.7	1.0	EDL	0.5	0.050	15-95	N-A-O
	193.7	1.0	EDL	0.2	0.020	2-20	Arg.-Hyd.
Cd	228.8	0.5	HCL	1.0	0.002	2-18	A-A-O
	326.1	1.0	HCL	400		180-800	
Pb	217.0	1.0	HCL	1.0	0.050	2.5-20	A-A-O
	283.3	0.5	HCL	2.6		7-50	
Cu	324.7	0.5	HCL	1.0	0.002	0.5-4.0	A-A-O
	222.6	1.0	HCL	40		40-180	
Fe	248.3	0.2	HCL	1.0	0.010	1.0-5.0	A-A-O
	386.0	0.2	HCL	16		36-145	

EDL: Electrode less Discharge Lamps, HCL: Hydrochloric acid, N-O-A: Nitrous Oxide Acetylene, A-A-O: Air Acetylene Oxidizing

Table 3 DPR target and intervention values of Metals for a standard soil/sediment (10% Organic Matter (OM) and 25% clay) mg/kg [7]

Metals	T_v	T_{int}	A	B	C
As	29	55	15	0.4	0.4
Ba	200	625	30	5	0
Cd	0.8	12	0.4	0.007	0.021
Cr	100	380	50	2	0
Cu	36	190	15	0.6	0.6
Hg	0.3	10	0.2	0.0024	0.0017
Pb	85	530	50	1	1
Ni	35	210	10	1	0
Zn	140	720	50	3	1.5

T_v and T_{int} are the standard (reference) target value and intervention value, respectively.

Table 4 Summary of the studied metal concentrations in the mobile and residual fractions

Metals	Cr	As	Cd	Pb	Cu	Fe
Mobile Conc.	65.49%	53.25 %	56.37%	41.17%	58.15%	1.86%
Residual Conc.	34.51%	46.76%	43.63%	58.83%	41.85%	98.14%
Origin	Anthropogenic	Anthropogenic	Anthropogenic	Lithogenic	Anthropogenic	Lithogenic

Mobile Conc. is the sum of the percentage metal concentrations in F_1 - F_4 of all the studied samples. Residual Conc. is the sum of the percentage metal concentrations in F_5 of all the studied samples.

Table 5 Physico-chemical parameters of the studied samples (soil and sediment)

Sample	Sand (%)	Silt (%)	Clay (%)	TOM (LOI) %	MC (%)	Ash Content (%)	pH value
SA	40	20	40	0.44	0.14	99.56	10.12
SB	33.54	25	41.56	0.62	0.17	99.38	9.73
SC	32.44	27.34	40.18	1.04	0.15	98.96	9.83

SA: Four mined samples (S_1 - S_4); SB: two sediment samples (S_5 - S_6); SC: two soil samples from the Iron ore processing/beneficiation yard (S_7 - S_8); MC: Moisture Content; TOM: Total Organic Matter; LOI: loss on ignition [19].

4. Results and Discussion

Table 4 shows a summary of metal concentrations in the mobile and residual fractions. It was noted that of all other fractions considered, F_1 (carbonate / acid-soluble) fraction had the least concentrations of metals in all the studied samples. The reason is that after the F_2 extraction using 1M $MgCl_2$, pH of 7, 0.5hrs, 20° C, the resultant residue is resistant to the F_1 extracting reagent ($CH_3COOH+CH_3COONa$) pH of 5,+5hrs, 20° C; therefore the oxides (F_3), the organics (F_4) and the residual (F_5) are increased while the carbonates (F_1) are reduced. The sums of the five fractions were in good agreement with the total digestion results with satisfactory recoveries of $\geq 100\%$ in all the samples; this indicates that the sequential extraction method is effective, reliable and reproducible. The results of the appraisal conducted to know the level of heavy metal contamination / pollution of NIOMCO Itakpe revealed that the area is only very slightly contaminated with the analysed metals: Cr, As, Cd, Pb and Cu. This implies that there might not be immediate danger of these metals pollution in the surrounding and in the food chain. Nevertheless since heavy metals have accumulative tendency, there might be serious danger of heavy metal pollution in the nearest future especially with increase in mining and beneficiation of iron ore in the company.

Out of the six metals analysed, four (Chromium, Arsenic, Cadmium and Copper) are from anthropogenic origin irrespective of some percentage of their concentrations from lithogenic source; this is because they occurred more in the mobile fractions than in the residual fractions of the sequential extraction scheme. The other two metals (Lead and Iron) are of lithogenic origin though

with some anthropogenic contributions. This is because they were seen more in the residual fractions of the scheme than in the mobile fractions. This means that Chromium, Arsenic, Cadmium and Copper are more bioavailable in the study area than Lead and Iron.

4.1. Characteristics of the Soil and Sediment Samples Studied

The particle size (percentage sand, silt and clay contents), total organic matter, moisture content, ash content and the pH of the studied samples (soil and sediment) are shown in Table 5.

4.2. Water Analysis

The concentrations of the six studied metals (Cr, As, Cd, Pb, Cu and Fe) in the two samples of water from river Pom-Pom (S_9 and S_{10}) are shown in Table 6.

As observed (Table 6) traces (0.000061 mg/l and 0.000065 mg/l) of Cu were seen from the two samples, S_9 (a) and (b), taken from the parent metals. These values are negligible compared to the standard acceptable values recommended in literature [20], [21] to determine the present level of contamination. Nevertheless, the suggested pH value for drinking water is between 6.5 and 8.5 [22]. The relatively high pH values (9.8-10), which is an indication of the alkalinity of the water samples, suggest an unpleasant smell or taste. This shows that the water is not safe for drinking.

Table 6 Concentrations of the six studied metals in samples of river Pom-Pom

Samples	pH	Elements Tested (mg/l)					
		Cr	As	Cd	Pb	Cu	Fe
S_9 (a)	10	ND	ND	ND	ND	0.000061	ND
S_9 (b)	10	ND	ND	ND	ND	0.000065	ND
S_{10} (a)	9.8	ND	ND	ND	ND	ND	ND
S_{10} (b)	9.8	ND	ND	ND	ND	ND	ND

ND: Not detected

5. Conclusion

The study on heavy metals pollution potentials in National Iron Ore Mining Company, Itakpe located in Kogi state has been investigated. The samples used for the study were obtained from the processing and smelting sections of the company. Out of the six metals analysed, four (Chromium, Arsenic, Cadmium and Copper) are from anthropogenic origin irrespective of some percentage of their concentrations from lithogenic source; this is because they occurred more in the mobile fractions than in the residual fractions of the sequential extraction scheme. The other two metals (Lead and Iron) are of lithogenic origin though with some anthropogenic contributions. This is because they were seen more in the residual fractions of the scheme than in the mobile fractions. This means that chromium, arsenic, cadmium and copper are more bioavailable in the study area than lead and iron. Conclusively, based on experimental studies on heavy metals, the findings indicated that human, animals and plants are exposed to toxic elements (metals and metalloids). The implications of these elements on health effects are challenging in so many ways, such as developmental retardation, several types of cancer, kidney damage, endocrine disruption, immunological, neurological effects and other disorders.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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